



January 21, 2016

Tom Moe USS Corporation P.O. Box 417 Mountain Iron, MN 55768

RE: Project: NPDES Line 3
Pace Project No.: 1259717

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on January 13, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Heather R Zika

Haller Zto

heather.zika@pacelabs.com

Project Manager

Enclosures

cc: Terri Sabetti, NTS





Pace Analytical www.pacelabs.com

315 Chestnut Street Virginia, MN 55792 (218) 742-1042

CERTIFICATIONS

Project: NPDES Line 3
Pace Project No.: 1259717

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792
Alaska Certification #MN01084
Arizona Department of Health Certification #AZ0785
Minnesota Dept of Health Certification #: 027-137-445
North Dakota Certification: # R-203

Wisconsin DNR Certification #: 998027470 WA Department of Ecology Lab ID# C1007 Nevada DNR #MN010842015-1 Oklahoma Department of Environmental Quality





SAMPLE SUMMARY

Project: NPDES Line 3
Pace Project No.: 1259717

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1259717001	WS-002 Scrubber Make-Up	Water	01/13/16 08:50	01/13/16 15:20
1259717002	WS-003 Thickener Overflow	Water	01/13/16 08:50	01/13/16 15:20

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SAMPLE ANALYTE COUNT

Project: NPDES Line 3
Pace Project No.: 1259717

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1259717001	WS-002 Scrubber Make-Up	EPA 200.7	MAR	3	PASI-V
		EPA 300.0	DMB	1	PASI-V
1259717002	WS-003 Thickener Overflow	EPA 200.7	MAR	3	PASI-V
		EPA 300.0	DMB	1	PASI-V



ANALYTICAL RESULTS

Project: NPDES Line 3
Pace Project No.: 1259717

Date: 01/21/2016 04:53 PM

Sample: WS-002 Scrubber Make	e-Up Lab ID:	1259717001	Collected	d: 01/13/16	8 08:50	Received: 01/	13/16 15:20 Ma	atrix: Water	
			Report						
Parameters	Results	Units	Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
200.7 MET ICP, Lab Filtered	Analytical	Method: EPA	200.7 Prepa	aration Meth	od: EP	A 200.7			
Calcium, Dissolved	93.6	mg/L	5.0	0.29	10	01/14/16 15:58	01/15/16 13:07	7440-70-2	
Magnesium, Dissolved	210	mg/L	5.0	0.67	10	01/14/16 15:58	01/15/16 13:07	7439-95-4	
Total Hardness, Dissolved	1100	mg/L	100	100 50.0 10		01/14/16 15:58	58 01/15/16 13:07		
300.0 IC Anions 28 Days	Analytical	Method: EPA	300.0						
Sulfate	831	mg/L	20.0	0.89	10		01/15/16 21:56	14808-79-8	
Sample: WS-003 Thickener Overflow	Lab ID:	1259717002	Collected	d: 01/13/16	8 08:50	Received: 01/	13/16 15:20 Ma	atrix: Water	
•	Lab ID:	1259717002 Units	Collected Report Limit	d: 01/13/16 MDL	08:50 DF	Received: 01/	13/16 15:20 Ma	atrix: Water CAS No.	Qual
Overflow	Results		Report Limit	MDL	DF	Prepared			Qual
• Overflow Parameters	Results	Units	Report Limit	MDL	DF	Prepared			Qual
Parameters 200.7 MET ICP, Lab Filtered	Results	Units Method: EPA 2	Report Limit 200.7 Prepa	MDL aration Meth	DF nod: EP	Prepared	Analyzed	CAS No.	Qual
Parameters 200.7 MET ICP, Lab Filtered Calcium, Dissolved	Results Analytical	Units Method: EPA 2 mg/L	Report Limit 200.7 Prepa	MDL aration Meth	DF nod: EP/	Prepared A 200.7 01/14/16 15:58	Analyzed 01/15/16 13:11 01/15/16 13:11	CAS No.	Qual
Parameters 200.7 MET ICP, Lab Filtered Calcium, Dissolved Magnesium, Dissolved	Results Analytical 682 49.8 1910	Units Method: EPA 2 mg/L mg/L	Report Limit 200.7 Prepa 5.0 5.0 100	MDL aration Meth 0.29 0.67	DF nod: EP/ 10 10	Prepared A 200.7 01/14/16 15:58 01/14/16 15:58	Analyzed 01/15/16 13:11 01/15/16 13:11	CAS No.	Qual

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QUALITY CONTROL DATA

Project: NPDES Line 3
Pace Project No.: 1259717

Date: 01/21/2016 04:53 PM

QC Batch: MPRP/6380 Analysis Method: EPA 200.7

QC Batch Method: EPA 200.7 Analysis Description: 200.7 MET Dissolved

Associated Lab Samples: 1259717001, 1259717002

METHOD BLANK: 281470 Matrix: Water

Associated Lab Samples: 1259717001, 1259717002

Reporting Blank Parameter MDL Result Limit Qualifiers Units Analyzed Calcium, Dissolved ND 0.50 0.029 01/15/16 12:12 mg/L Magnesium, Dissolved mg/L ND 0.50 0.067 01/15/16 12:12

LABORATORY CONTROL SAMPLE: 281471 Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Calcium, Dissolved 50 49.5 99 85-115 mg/L Magnesium, Dissolved 50 49.2 98 85-115 mg/L

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 281472 281473 MSD MS 1259385001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits **RPD** RPD Qual Calcium, Dissolved mg/L 29.1 50 50 79.1 0.08 100 102 70-130 20 Magnesium, Dissolved mg/L 71.9 50 50 122 123 99 101 70-130 20

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 281474 281475 MS MSD MS 1259528003 MSD MS Spike Spike MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits **RPD** RPD Qual Calcium, Dissolved 50 25.0 50 77.0 77.0 104 104 70-130 0 20 mg/L 50 Magnesium, Dissolved 11.9 50 63.1 63.8 102 104 70-130 20 mg/L 1

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALITY CONTROL DATA

Project: NPDES Line 3
Pace Project No.: 1259717

QC Batch: WETA/15403 Analysis Method: EPA 300.0

QC Batch Method: EPA 300.0 Analysis Description: 300.0 IC Anions

Associated Lab Samples: 1259717001, 1259717002

METHOD BLANK: 281901 Matrix: Water

Associated Lab Samples: 1259717001, 1259717002

ParameterUnitsBlank Reporting ResultReporting LimitMDLAnalyzedQualifiersSulfatemg/LND2.00.08901/15/16 13:53

LABORATORY CONTROL SAMPLE: 281902

Date: 01/21/2016 04:53 PM

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Sulfate mg/L 50 49.9 100 90-110

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 281903 281904

MS MSD

1259752001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits RPD RPD Qual Sulfate 90-110 0 20 mg/L 28.8 50 50 81.1 81.0 105 104

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 281905 281906

MS MSD MS MSD MS 1259764001 Spike Spike MSD % Rec Max Limits RPD Parameter Units Result Conc. Conc. Result Result % Rec % Rec RPD Qual Sulfate ND 50 50 51.2 51.2 101 101 90-110 0 20 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALIFIERS

Project: NPDES Line 3
Pace Project No.: 1259717

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

Date: 01/21/2016 04:53 PM

PASI-V Pace Analytical Services - Virginia

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: NPDES Line 3
Pace Project No.: 1259717

Date: 01/21/2016 04:53 PM

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1259717001	WS-002 Scrubber Make-Up	EPA 200.7	MPRP/6380	EPA 200.7	ICP/4876
1259717002	WS-003 Thickener Overflow	EPA 200.7	MPRP/6380	EPA 200.7	ICP/4876
1259717001	WS-002 Scrubber Make-Up	EPA 300.0	WETA/15403		
1259717002	WS-003 Thickener Overflow	EPA 300.0	WETA/15403		

SSU CLIENT NAME, ADDRESS, PHONE#: RELINQUISHED BY: RECEIVED BY: RELINQUISHED BY: PROFILE NO: 146 PROJECT: Pace Analytical® NPDES-LINE 3 WS-003 Thickner WS-002 Scrubber CUSTODY SEALS INTACT: Make-up Overflow BO# 140 315 Chestnut Street P.O. Box 1212 Virginia, MN 55792 218-742-1042 SAMPLER, DATE: INVOICE TO: Pace Analytical Services. Inc. ξŠ のがど言言 DATE: 11316 RELINQUISHED BY: DATE: 17376 RECEIVED BY: SAMPLE COLLECTION DATE START DATE: 73-16 ĕ RECEIVED BY: 1-13-16 1-13-16 END DATE: L'audinature 08180 PERMIT REQ.: MN0057207 08:80 START TIME: SAMPLE COLLECTION TIME 08,80 08,50 END TIME: COMP SAMPLE TYPE GRAB SAMPLES RECEIVED ON ICE: DATE: TIME: DATE: DATE: ē CLIENT: USS CORP No#:1259717 MATRIX RECEIVED FOR LAB BY: 2 GENERAL - NO PRES. RECEIVED FROM PACE SAMPLE LOCK-UP RELINQUISHED TO PACE SAMPLE LOCK-UP: N **NUTRIENTS - H2S04** Æ Due Date: 01/27/16 3Y RECORD METALS - HN03 40ML VIALS -HCL 1LT AMBER - HCL Š 500 ml - Zn Acetate SPECIAL INSTRUCTIONS: ANALYSIS: LAB FILTER: SO₄, Ca, Mg, Hard. Calc. LAB FILTER: SO4, Ca, SAMPLE TEMPERATURE: MINNTAC LAB

Mg, Hard. Calc.

TIME:

DATE

DATE :IME

DATTE # 1-13-

15:20

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Face Analytical*

hold, incorrect preservative, out of temp, incorrect containers)

Document Name:

Sample Condition Upon Receipt Form

Document No.: F-VM-C-001-Rev.09 Document Revised: 23Feb2015

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Issuing Authority:

Pace Virginia, Minnesota Quality Office

Sample Condition Client Name: Upon Receipt	<		Projec	"# WO#:1259717
Courier: Fed Ex UPS Commercial Pace	USPS □Othe	/-	_ Client	
Tracking Number:		· · · · · · · · · · · · · · · · · · ·		- 1259717
Custody Seal on Cooler/Box Present? Yes	ŽNo	Seals	Intact?	□Yes ☑No Optional: Proj. Due Date: Proj. Name:
Packing Material: Bubble Wrap Bubble B	ags 🔼 i	None	Other:	Temp Blank? 🖊 Yes 🗆 No
Thermometer Used: 🚺 140792808	•			,
Cooler Temp Read °C: 2. Cooler Temp Read °C: Correction Fac	Corrected	-	2.4	Biological Tissue Frozen? Yes No NA Mod Initials of Person Examining Contents:
Chain of Custody Present?	[Z]Yes	□No	□N/A	1.
Chain of Custody Filled Out?	∠ Yes	□No		. 2.
Chain of Custody Relinquished?	Z]Yes	□No	□N/A	3.
Sampler Name and Signature on COC?	Yes	□No	□N/A	4.
Samples Arrived within Hold Time?	✓ Yes	□No	□N/A	5.
Short Hold Time Analysis (<72 hr)?	☐Yes	ZNo	□N/A	6.
Rush Turn Around Time Requested?	Yes	ØN₀	□n/a	7.
Sufficient Volume?	ØŶes	□No	□N/A	8.
Correct Containers Used?	✓Yes	□No	□n/a	9.
-Pace Containers Used?	∕— ∑Yes	□No	□n/a	
Containers Intact?	Z Yes	□No	□N/A	10.
Filtered Volume Received for Dissolved Tests?	☐Yes	□No	□/nya	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	Yes	□No		12. Wite it sectation is visible in the dissolved containers.
-Includes Date/Time/ID/Analysis Matrix: CV			U14/A	
All containers needing acid/base preservation will be checked and documented in the pH logbook.	Yes	□No	Z)n/a	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	Yes	∐No	J-N/A	13.
Headspace in VOA Vials (>6mm)?	Yes	□No	Øn/a	14.
Trip Blank Present?	□Yes	□No	ØN/A	15.
Trip Blank Custody Seals Present? Pace Trip Blank Lot # (if purchased):	Yes	□No	(Z)N/A	
CLIENT NOTIFICATION/RESOLUTION Person Contacted:			D	Field Data Required? Yes No
Comments/Resolution:				
	-			
FECAL WAIVER ON FILE Y N Project Manager Review:	1 25	7		E WAIVER ON FILE Y N
Note: Whenever there is a discrepancy affecting North Carolina old, incorrect preservative, out of temp, incorrect containers)	compliance :	samples, a	copy of this	s form will be sent to the North Carolina DEHNR Certification Office (i.e. out of